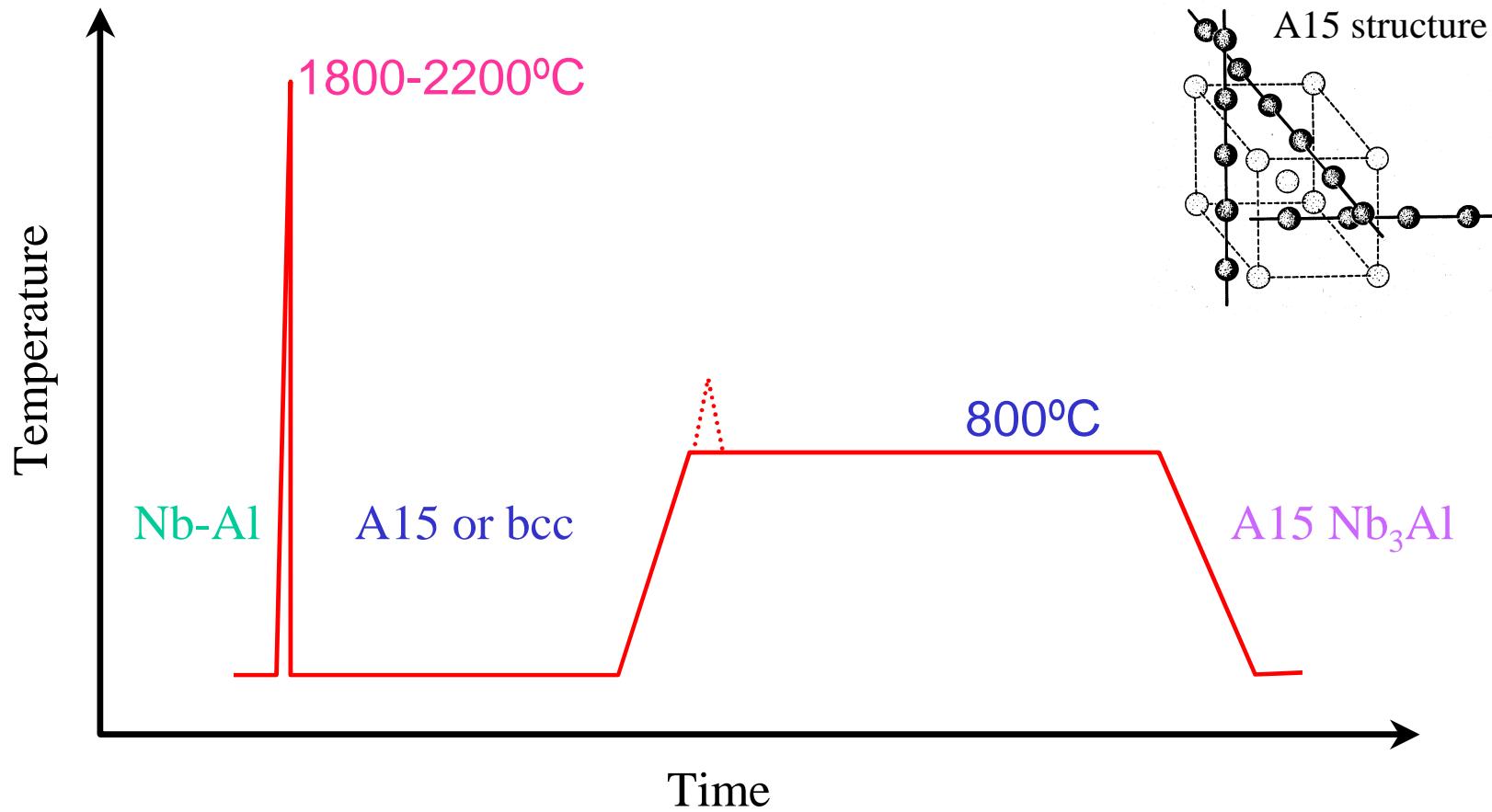


Phase evolution and transformation during thermal processing of Nb-Al superconductors

Florin Buta, Michael D. Sumption, Edward W. Collings

LASM, MSE, Ohio State University

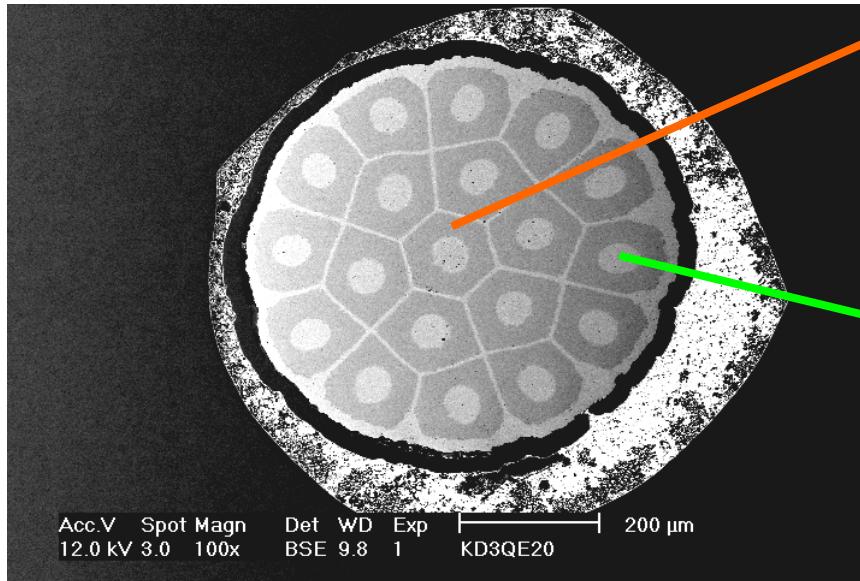
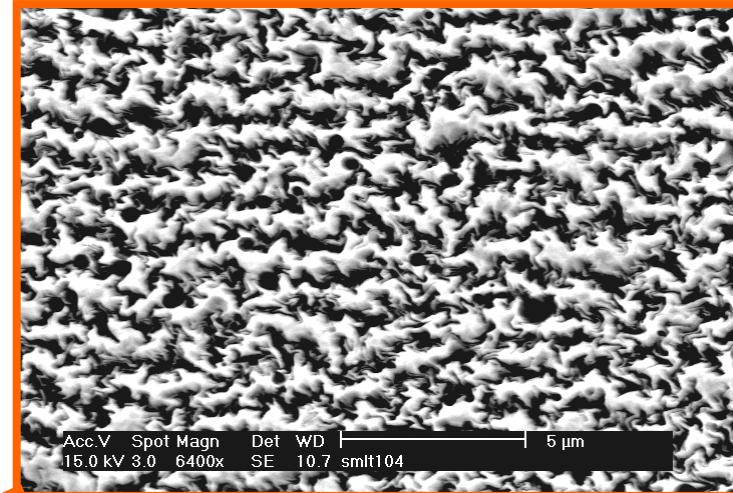
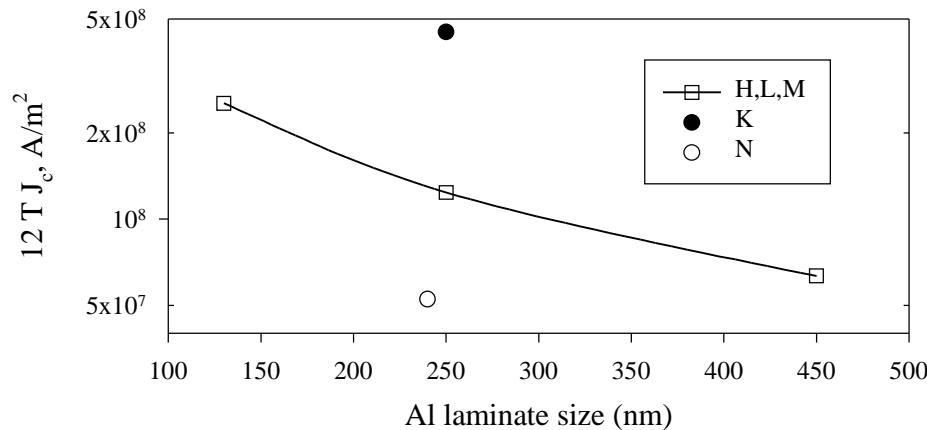
RHQT Processing



Rapid Heating and Quenching

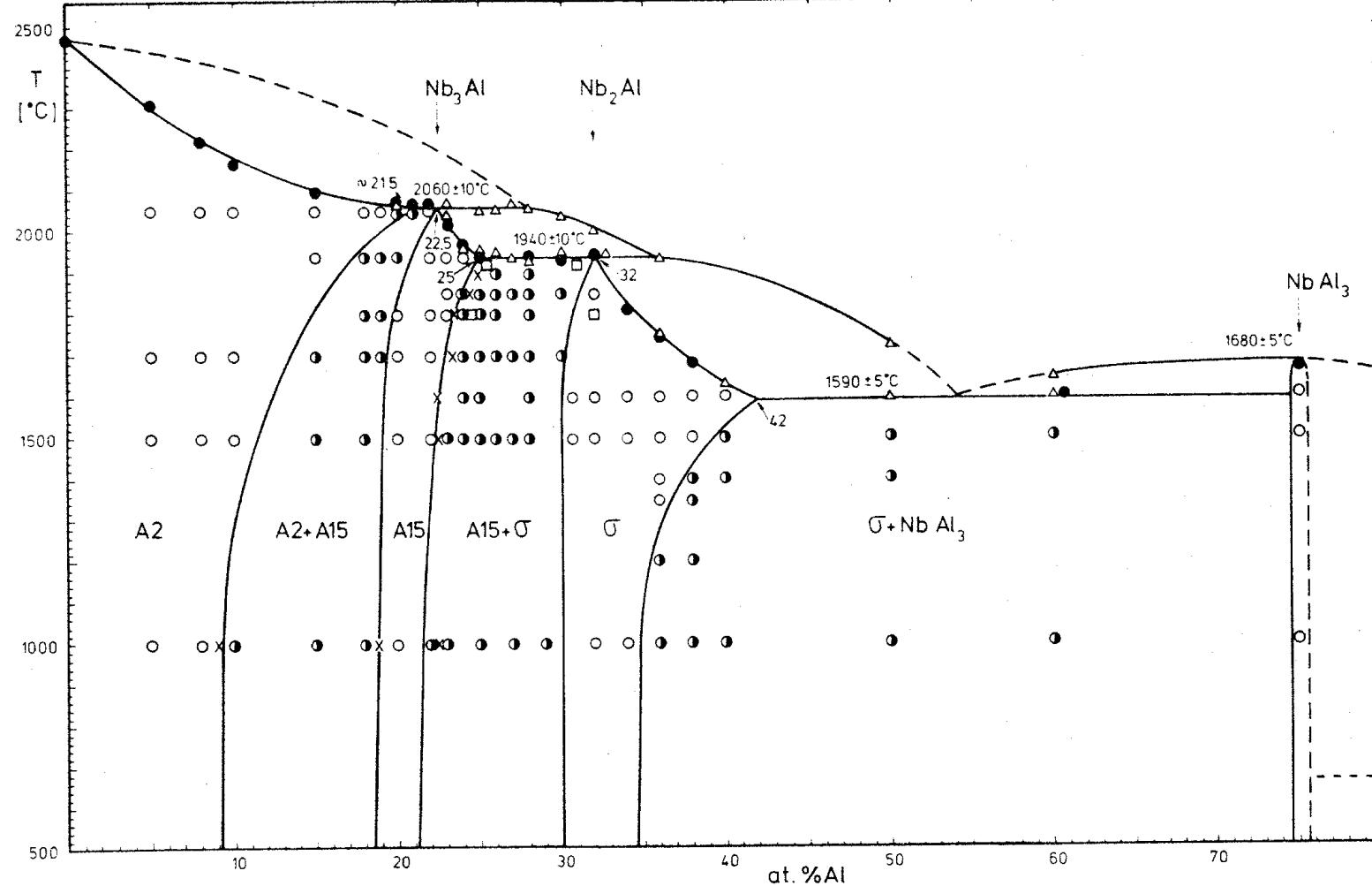
Transformation

Precursor composite wires - laminate size



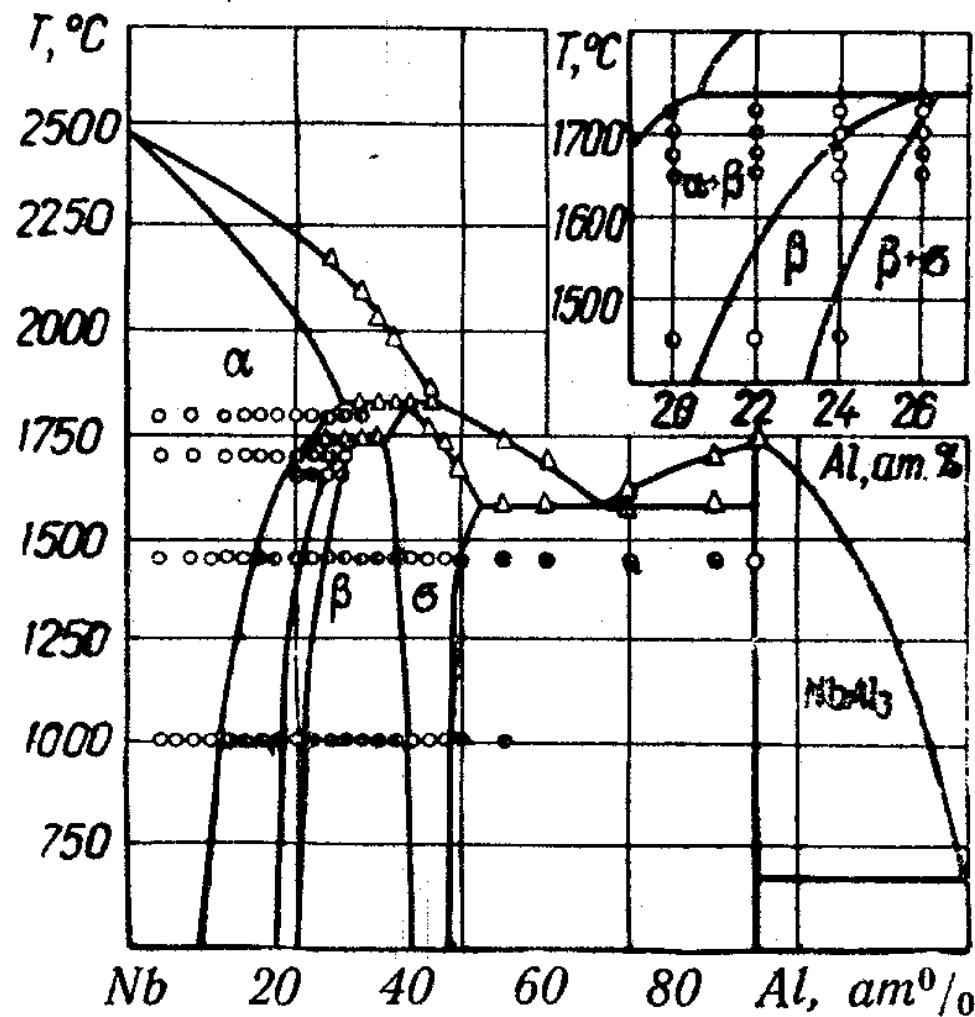
pure Niobium

Nb-Al equilibrium



Jorda et al, J. Less Common Metals 75 (1980) 227-239.

Nb-Al equilibrium



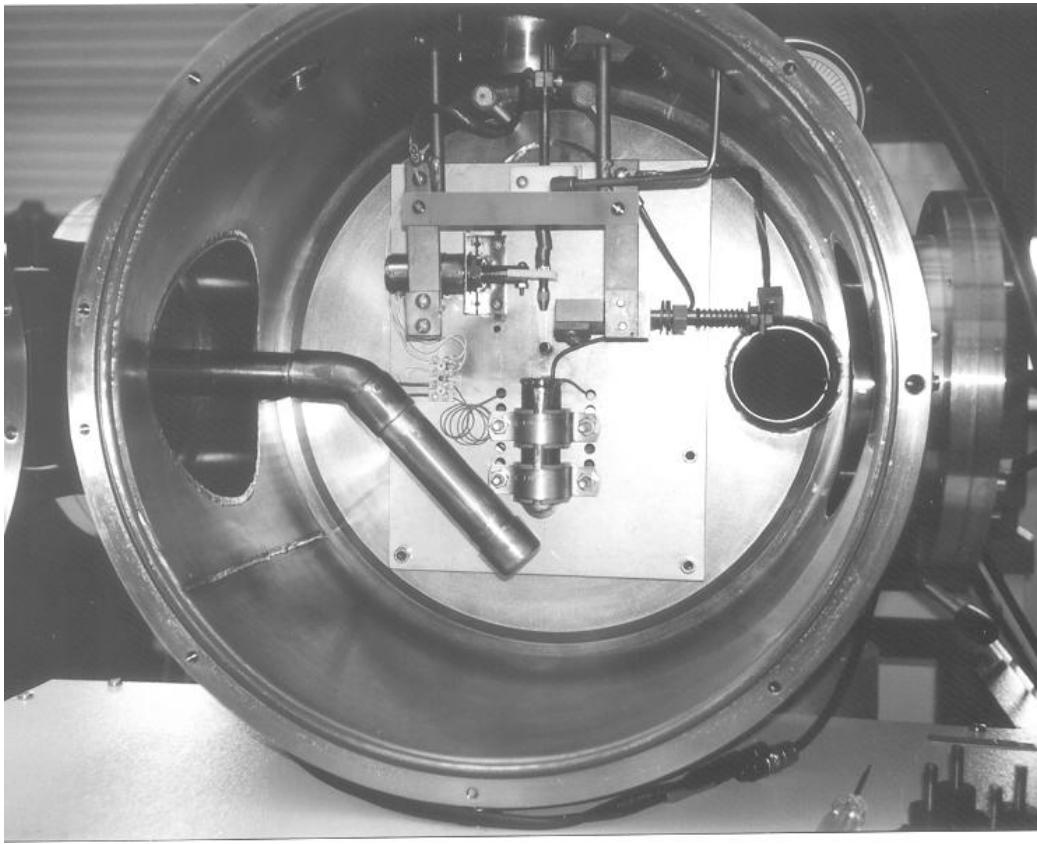
Svechnikov et al, Metallofizika 22 (1968) 54-60.

Sample specifications

Nb/Al volumic ratio	Overall composition (at% Al)	Al laminate size (nm)
3.00	26.8	240
3.30	25.0	146
3.30	25.0	210
3.75	22.7	127
3.75	22.7	200

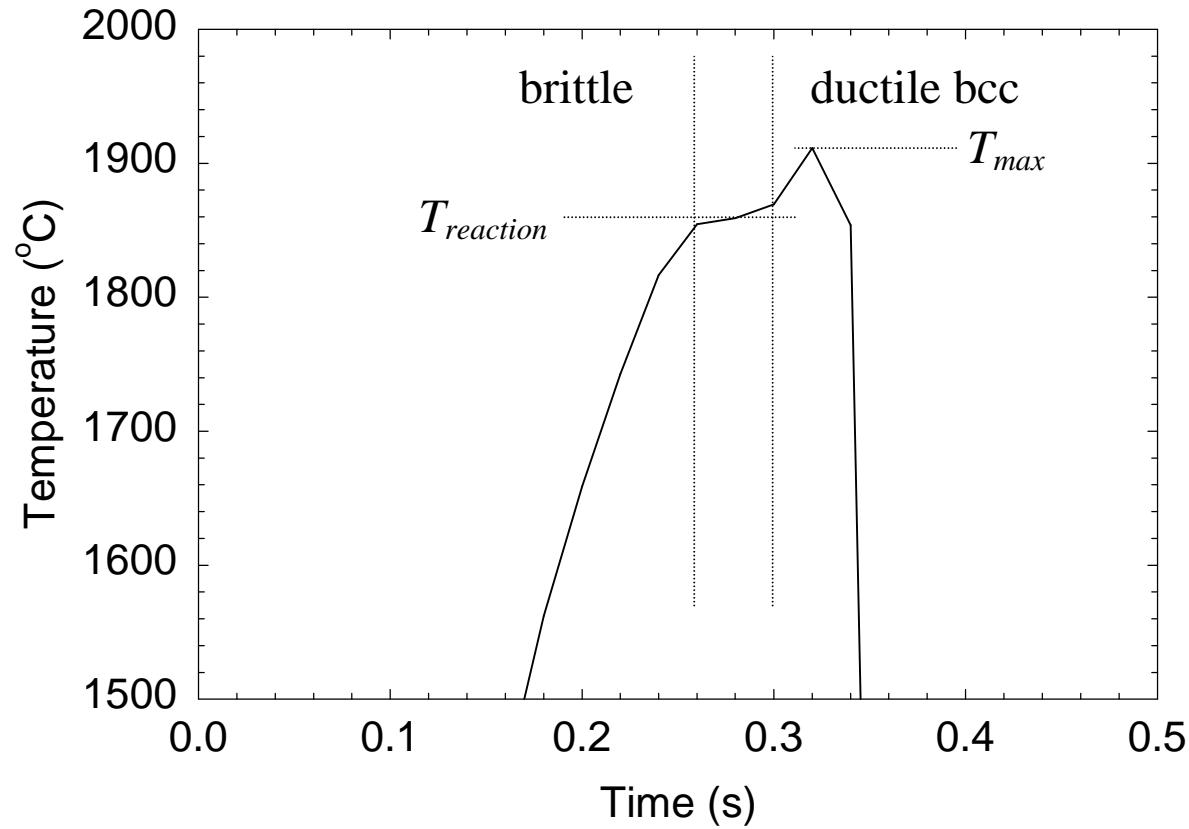
- Samples rapidly heated to different T_{\max} and quenched;
- Temperature recorded during rapid heating;
- XRD on tape pressed samples (to maximize signal);
- Back-scattering SEM on cross-sections of round wires.

Short sample quenching apparatus



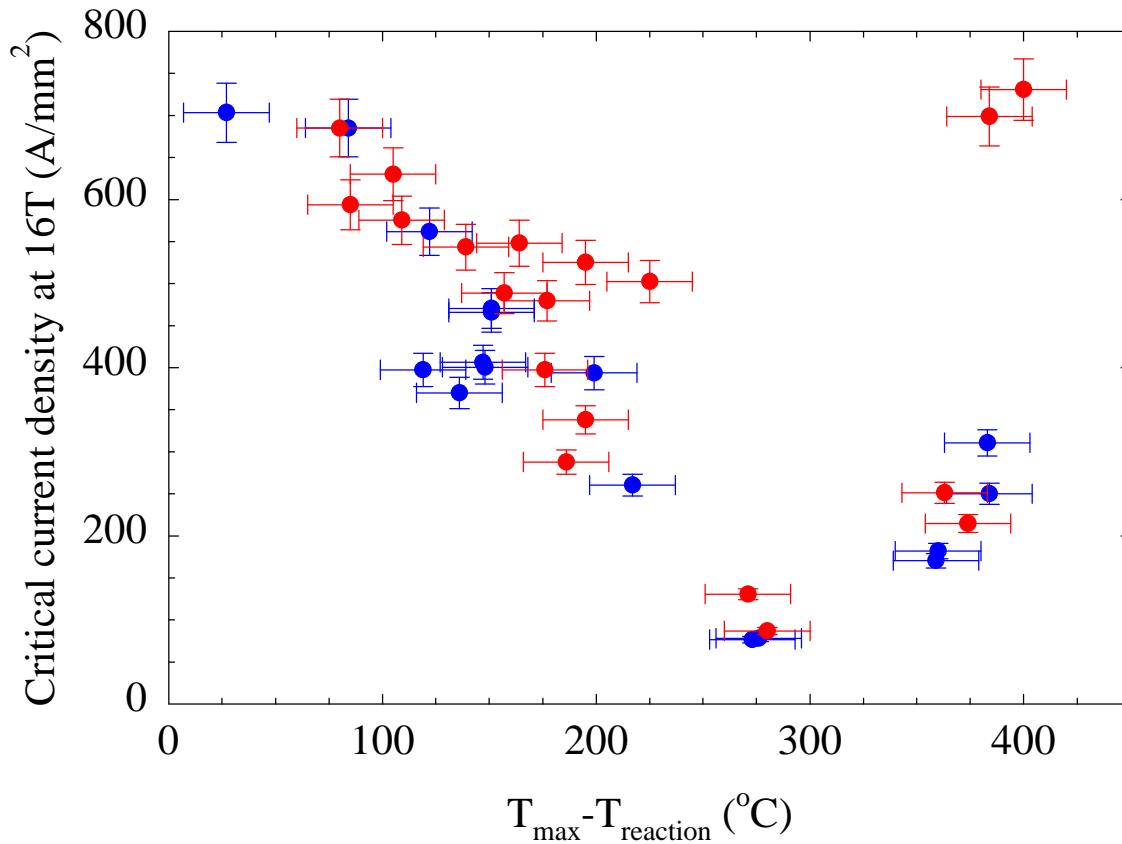
Electrical self-heating in vacuum + quenching in liquid Ga.

bcc formation reaction

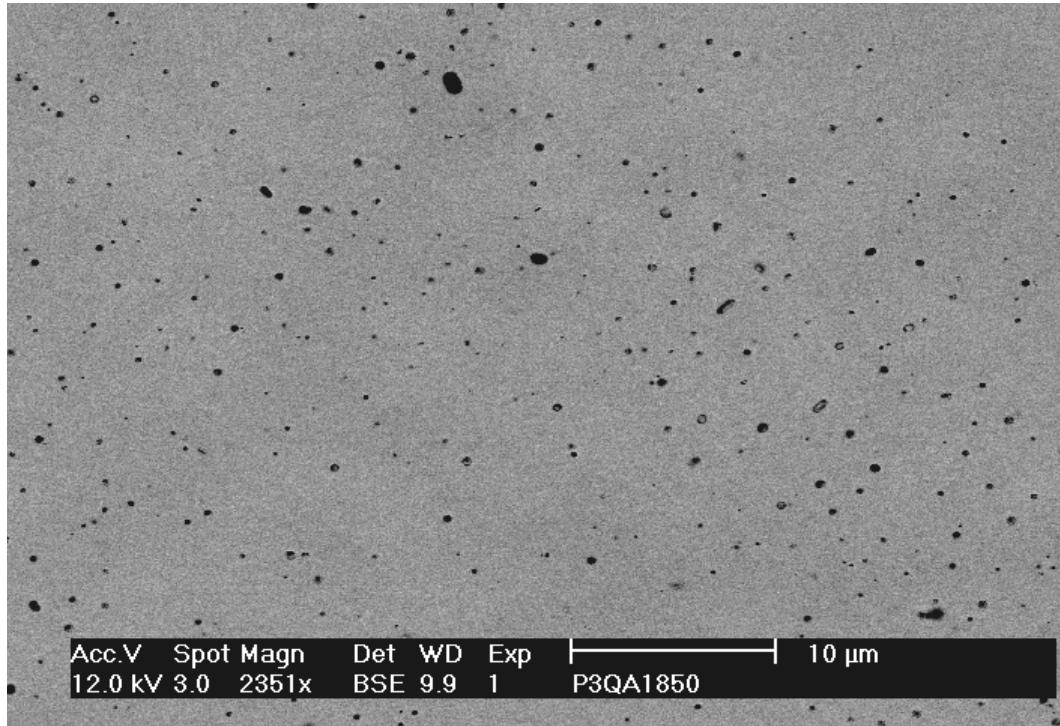


Endothermic reaction at rapid heating.

J_c function of T_{max} - $T_{reaction}$



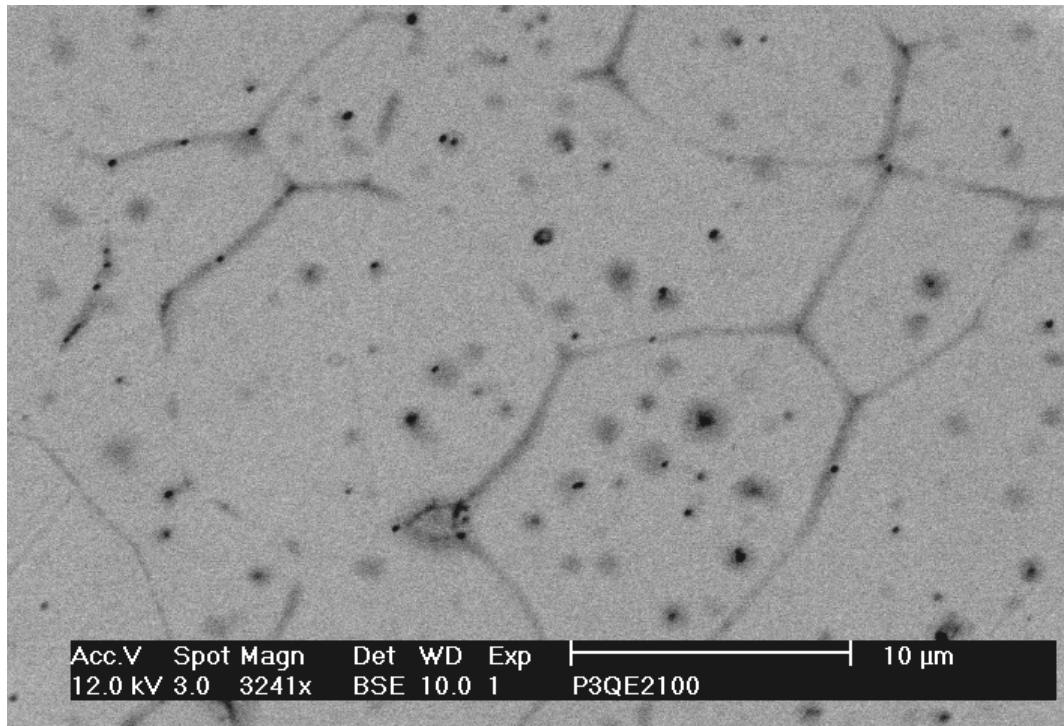
Microstructure at ~80°C above $T_{reaction}$



(22.7 at% Al)

Porosity in homogeneous material.

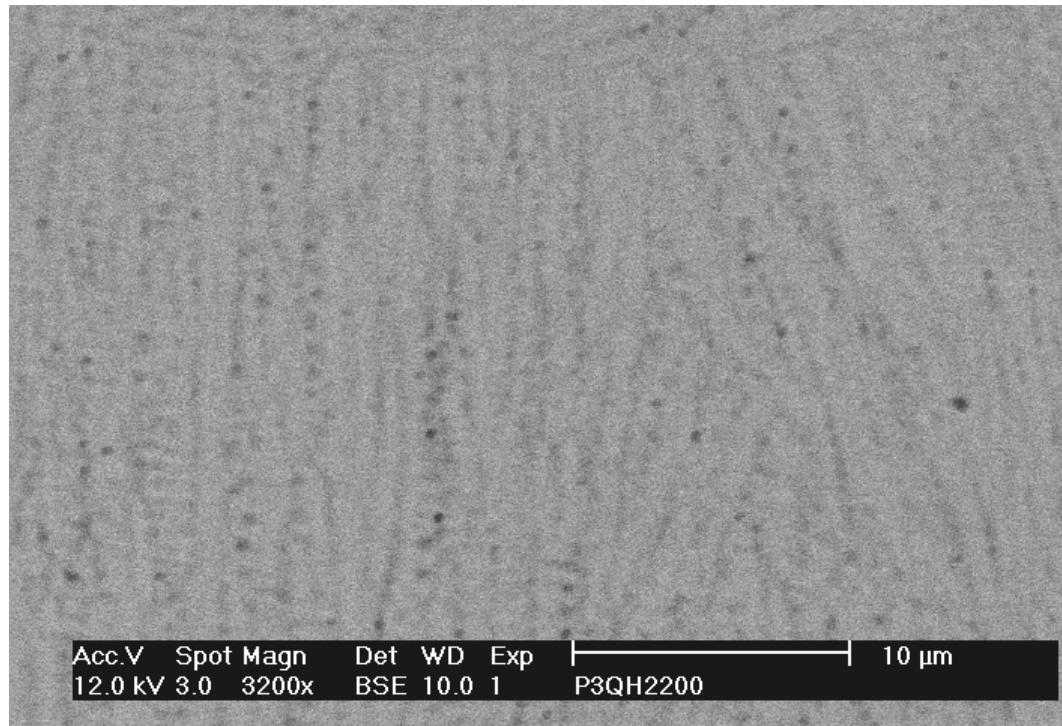
Microstructure at $\sim 230^{\circ}\text{C}$ above T_{reaction}



(22.7 at% Al)

Melting at grain boundaries and around pores.

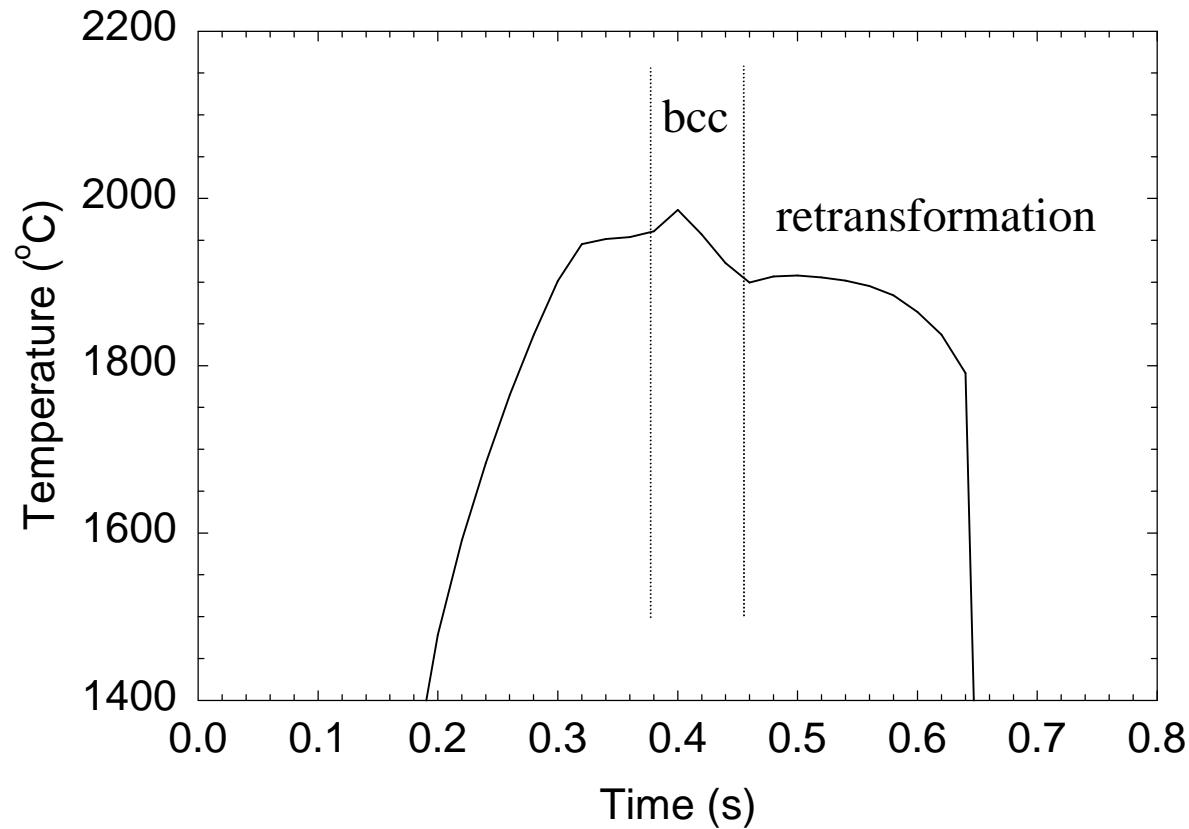
Microstructure at ~380°C above $T_{reaction}$



(22.7 at% Al)

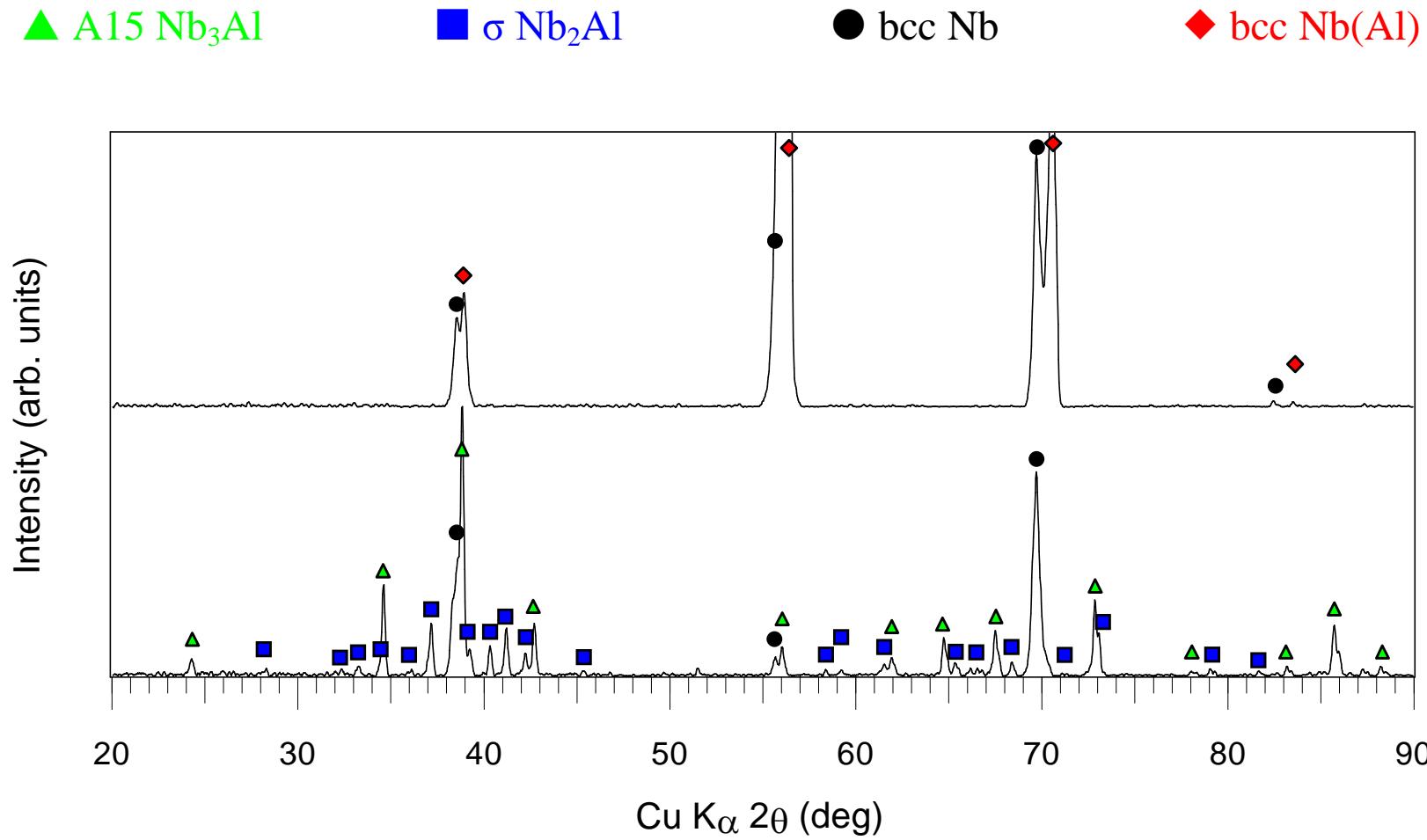
Dendritic structure of rapidly solidified liquid (+large pores).

Retransformation from bcc



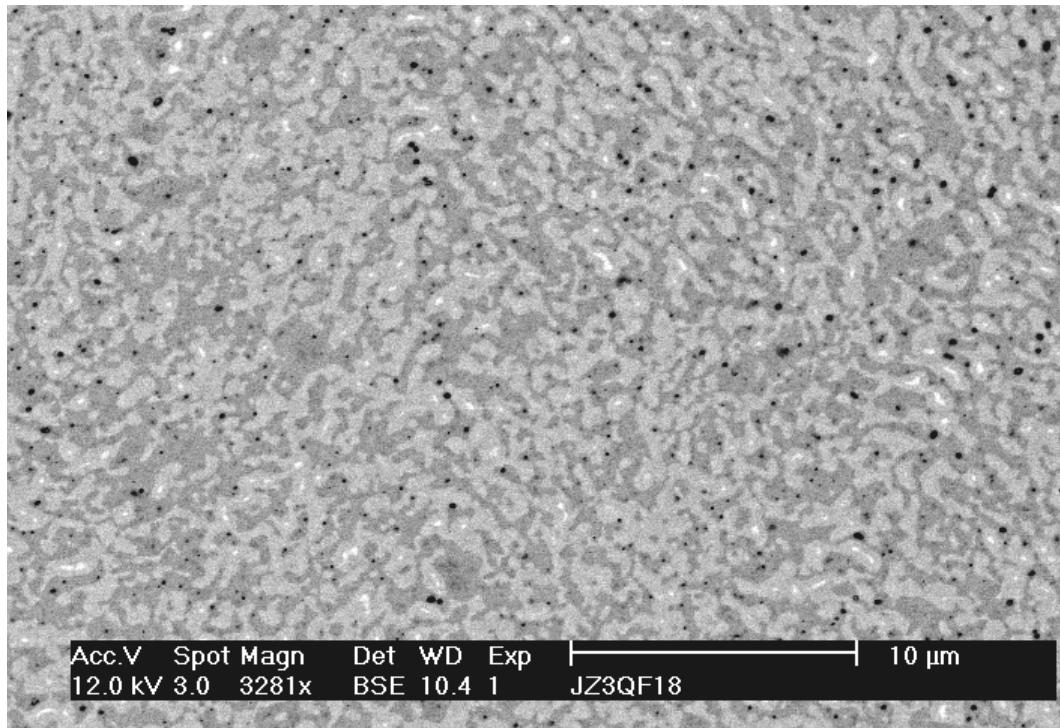
If samples not quenched immediately after heating is stopped, bcc retransforms.

Phase evolution at 25at%Al



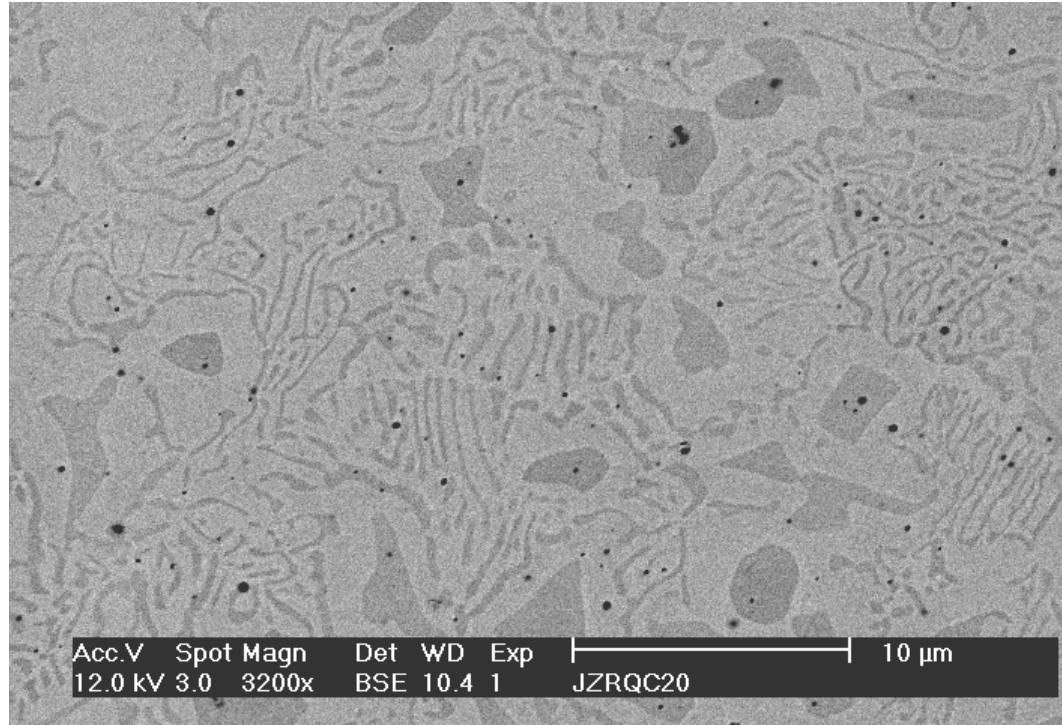
A15 and σ -phase combine to form bcc Nb(Al) solid solution.

Microstructure before reaction, at 25at%Al



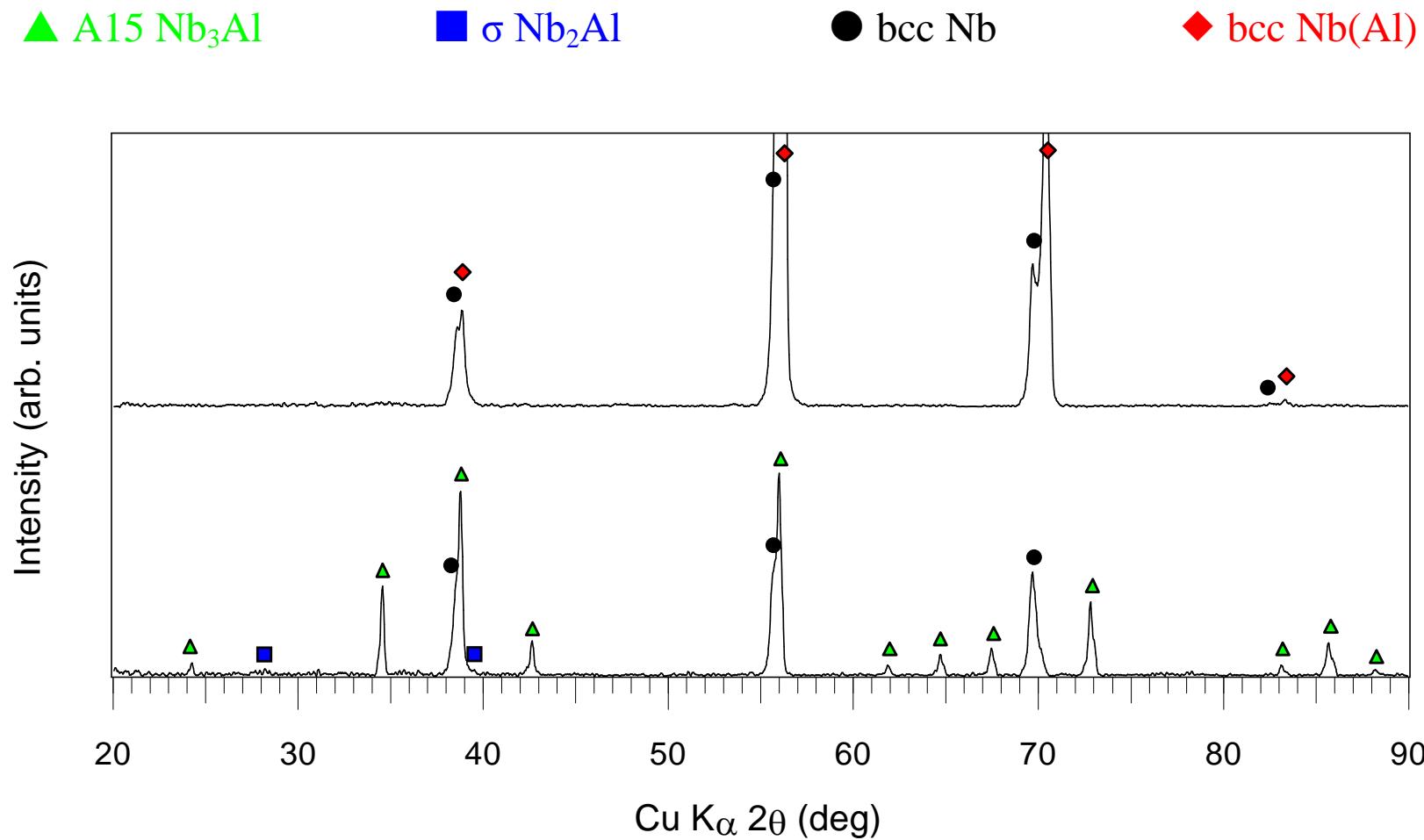
Laminar structure of the precursor jelly-roll maintained.

Microstructure after retransformation, at 25at%Al



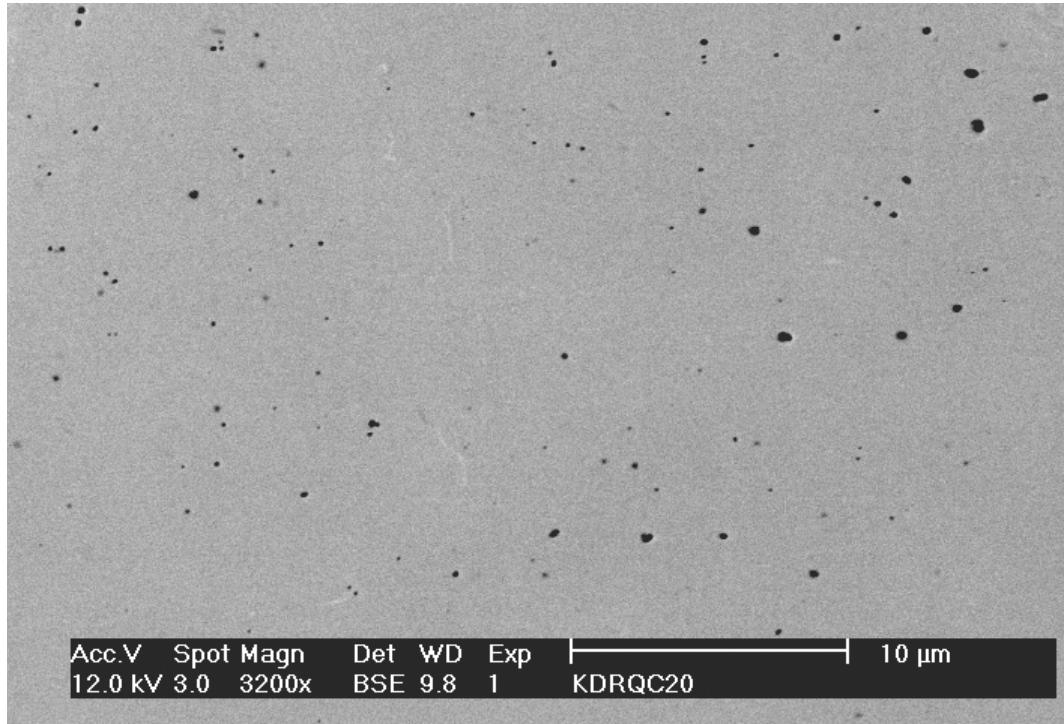
Eutectoidal decomposition of bcc to A15 and σ -phase.

Phase evolution at 22.7at%Al



A15 transforms to bcc Nb(Al) solid solution.

Microstructure after retransformation, at 22.7at%Al



Massive transformation from bcc to A15.

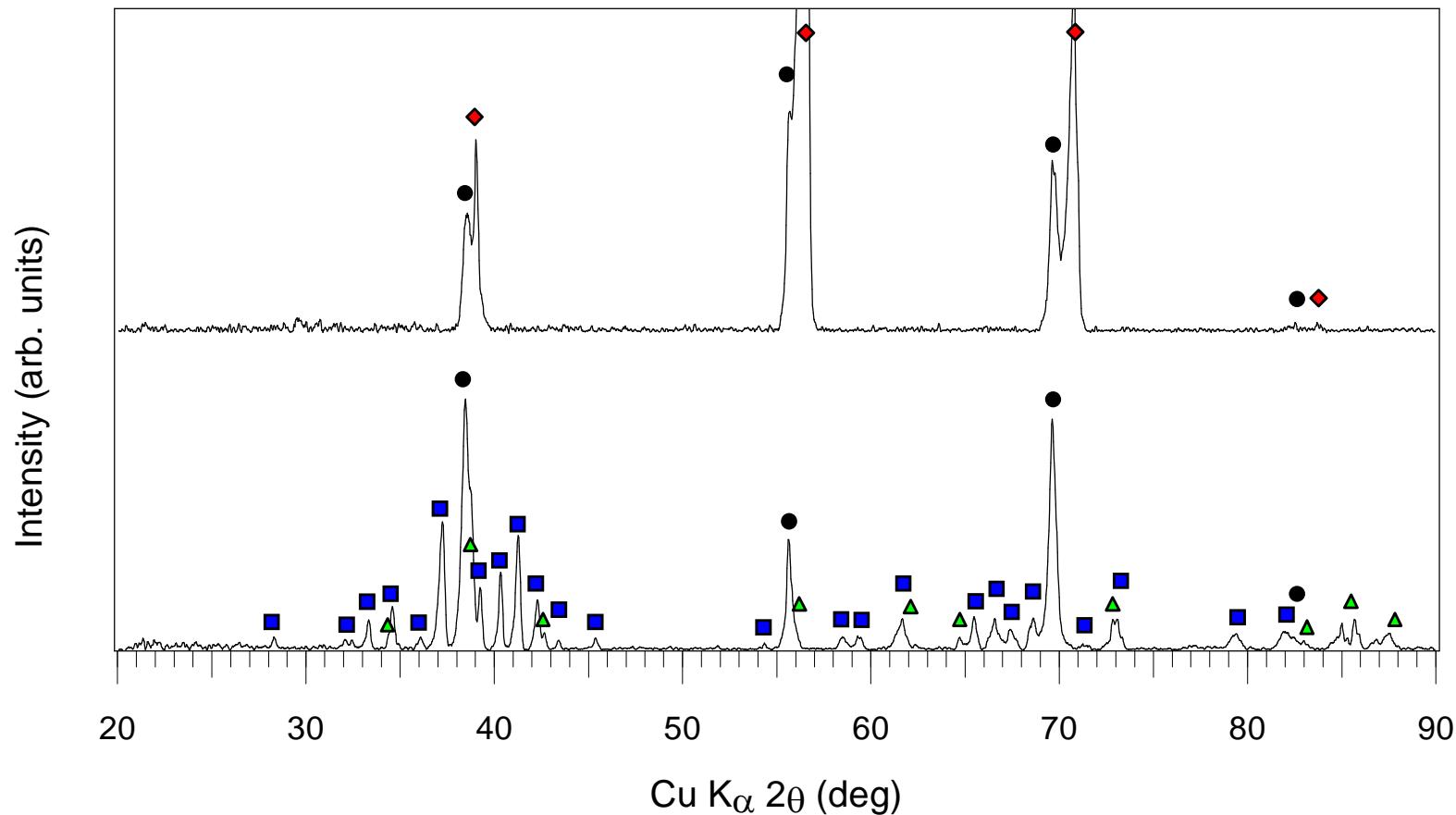
Phase evolution at 26.8at%Al

▲ A15 Nb₃Al

■ σ Nb₂Al

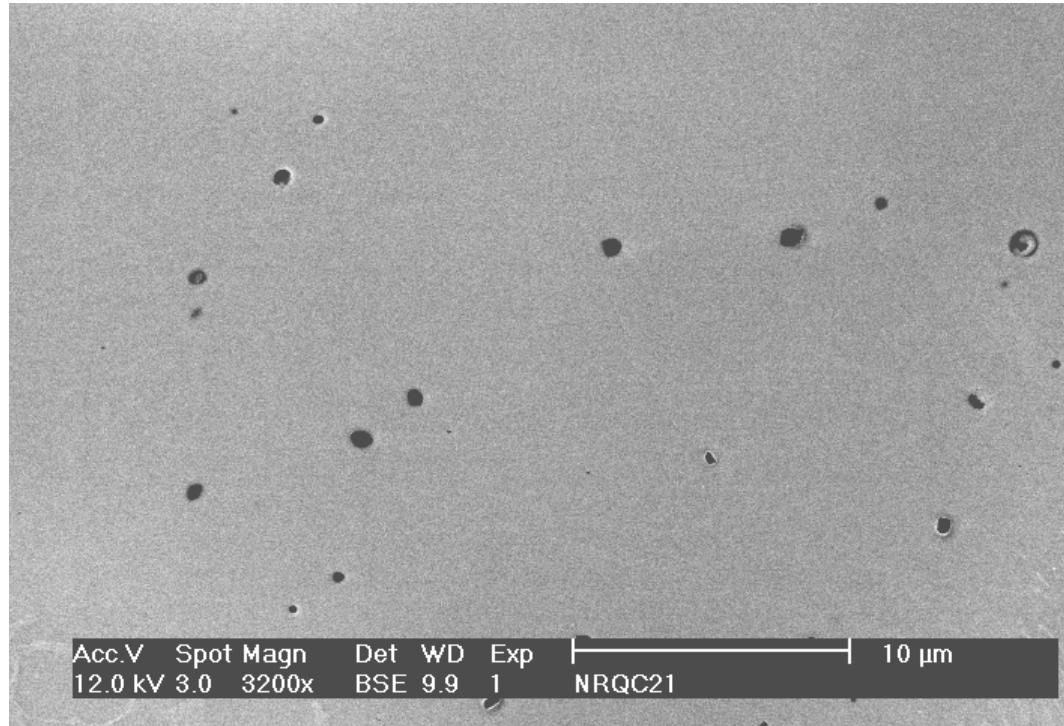
● bcc Nb

◆ bcc Nb(Al)



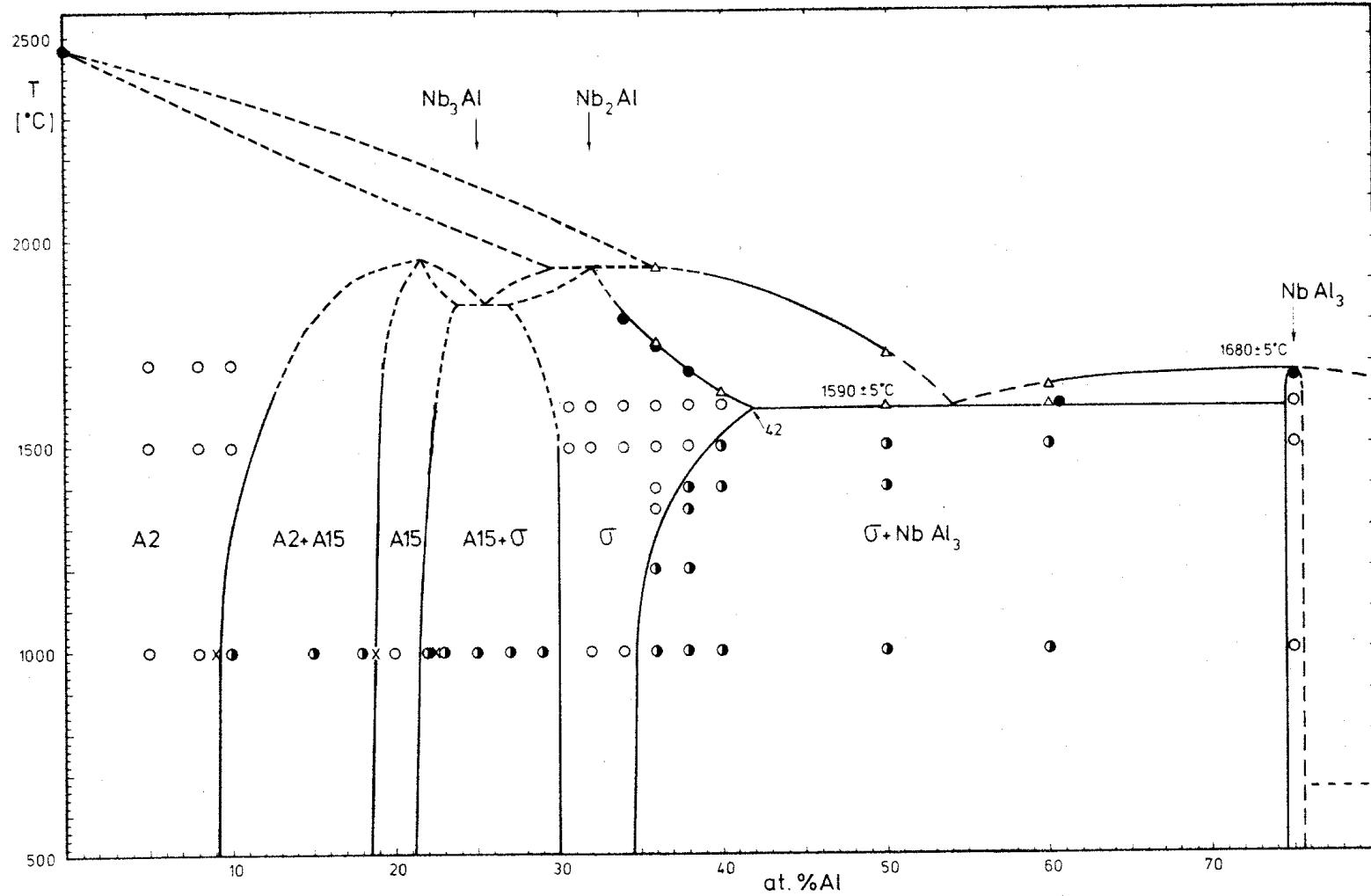
σ -phase transforms to Nb(Al) solid solution.

Microstructure after retransformation, at 26.8at%Al

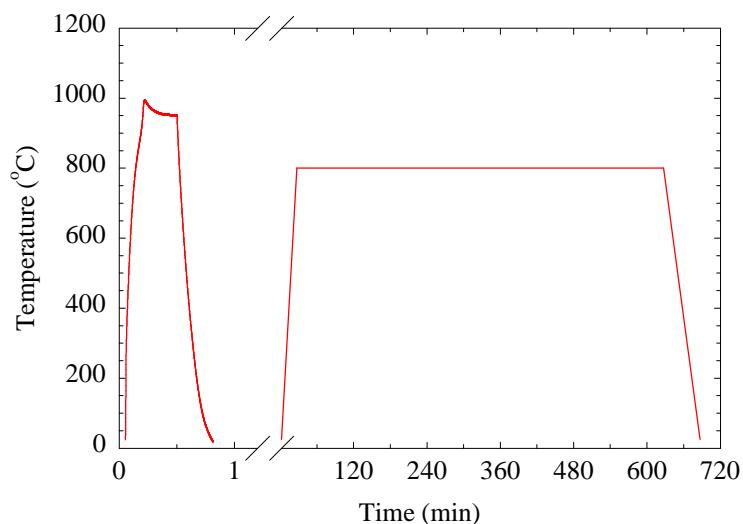
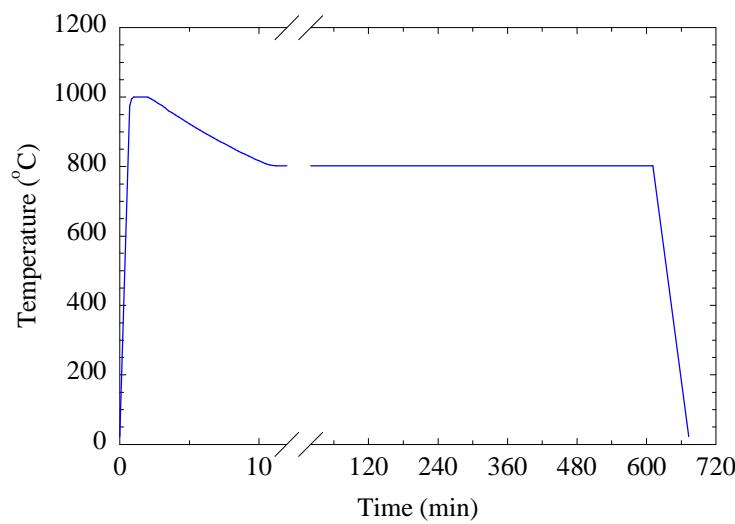
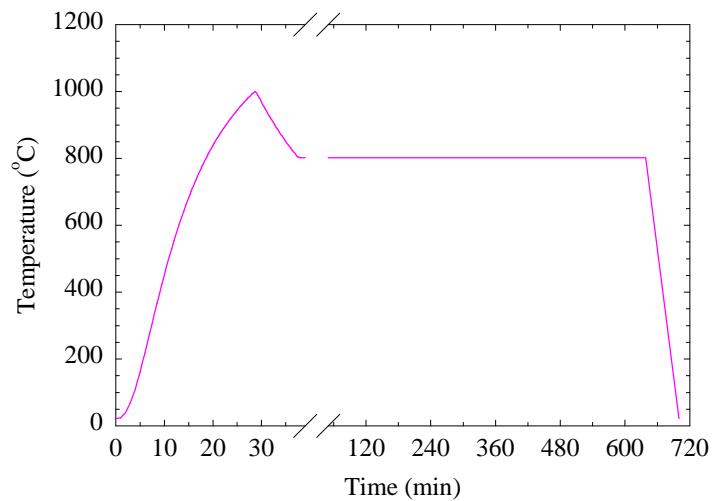
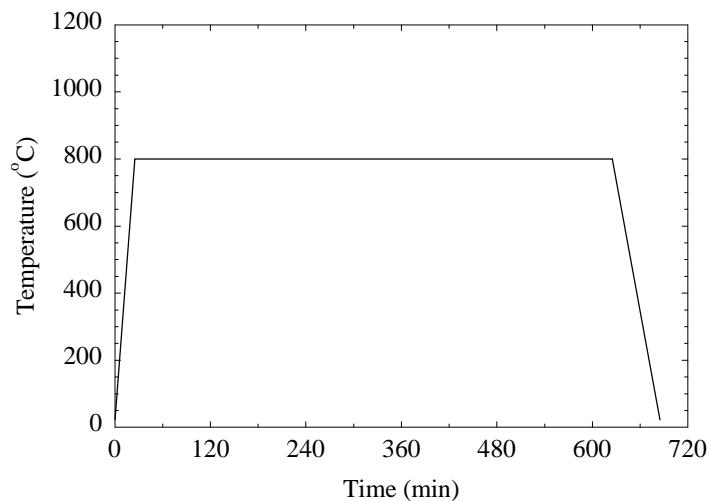


Massive transformation from bcc to σ -phase.

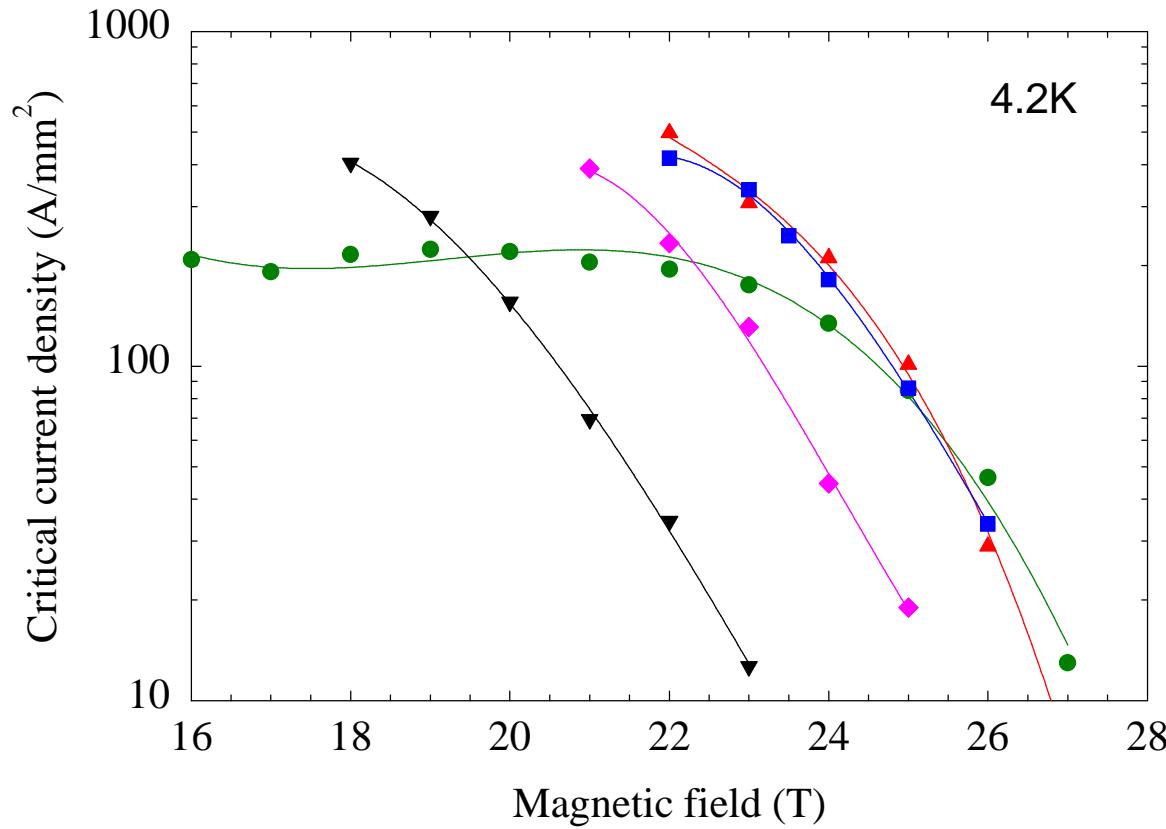
Nb-Al equilibrium ?



Transformation heat treatments

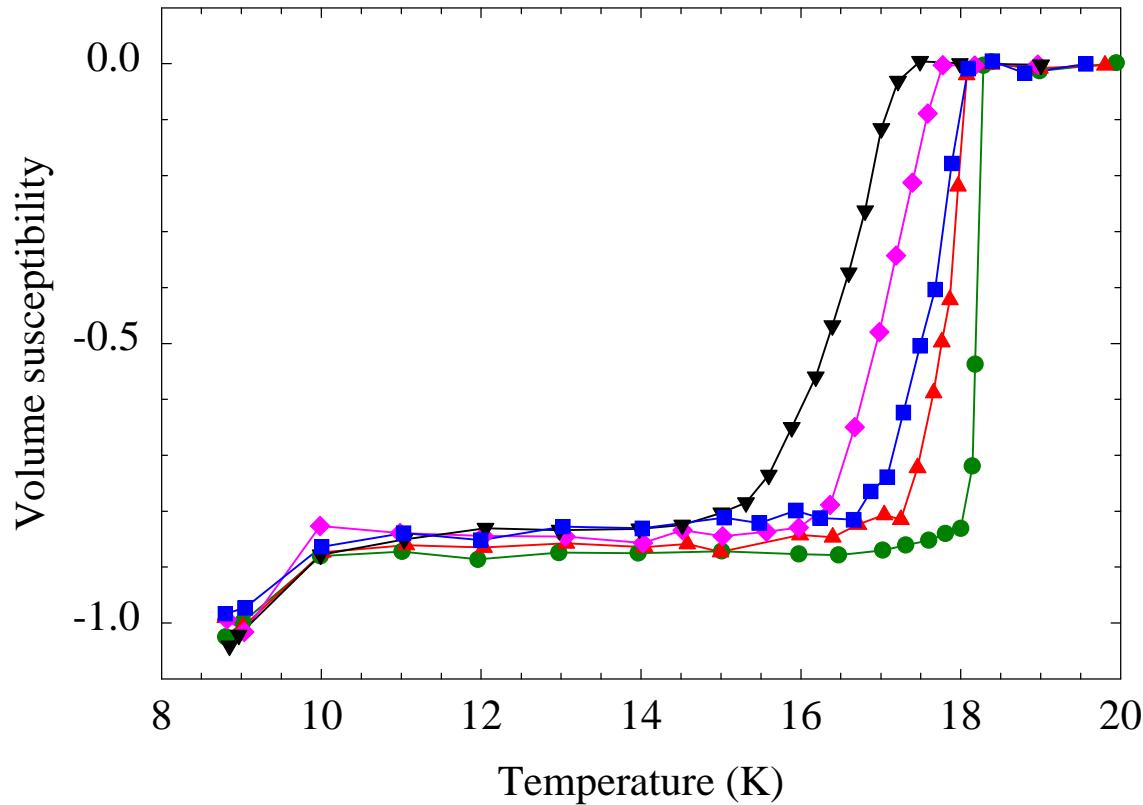


Critical current densities for 25at%Al samples



- A15-quenched + 800°C/10h
- ▼ bcc-quenched + 800°C/10h
- ◆ bcc-quenched + furnace heating to 1000°C in 30min + 800°C/10h
- bcc-quenched + insert in furnace at 1000°C, hold for 2min + 800°C/10h
- ▲ bcc-quenched + ohmic pulse 1000°C/30s + 800°C/10h

Magnetic susceptibility for 25at%Al samples

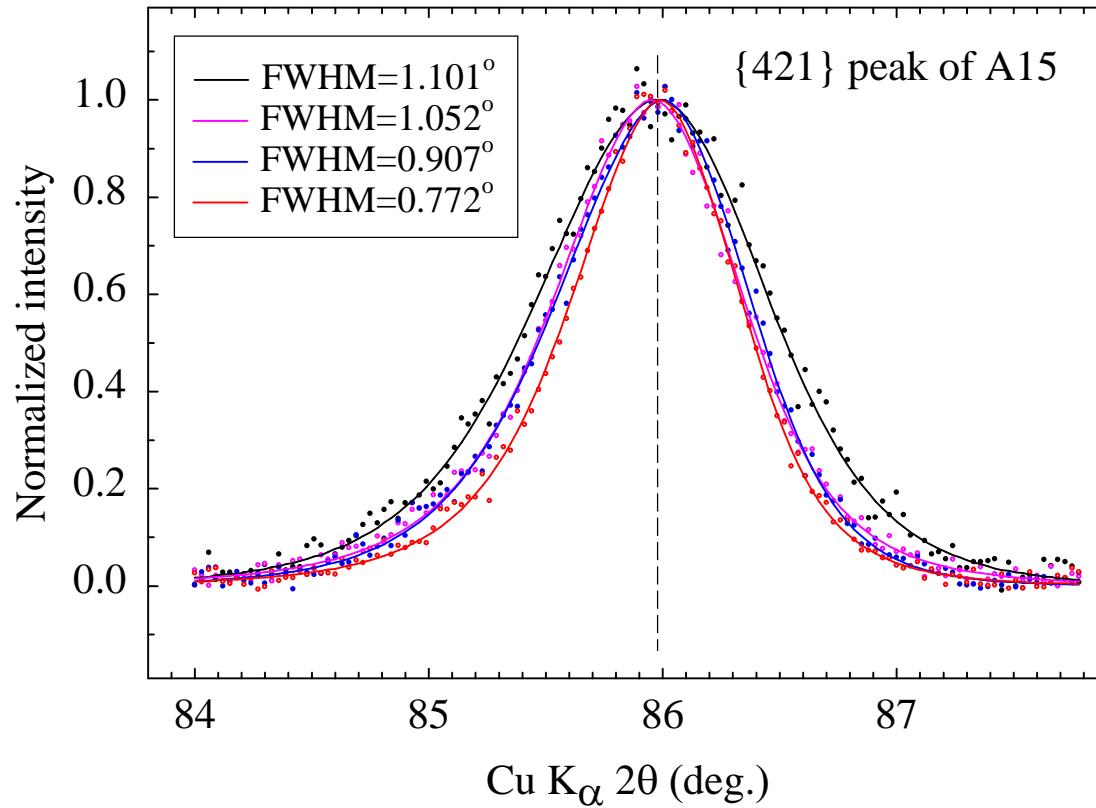


- A15-quenched + 800°C/10h
- ▼ bcc-quenched + 800°C/10h
- ◆ bcc-quenched + furnace heating to 1000°C in 30min + 800°C/10h
- bcc-quenched + insert in furnace at 1000°C, hold for 2min + 800°C/10h
- ▲ bcc-quenched + ohmic pulse 1000°C/30s + 800°C/10h

Summary of magnetic properties

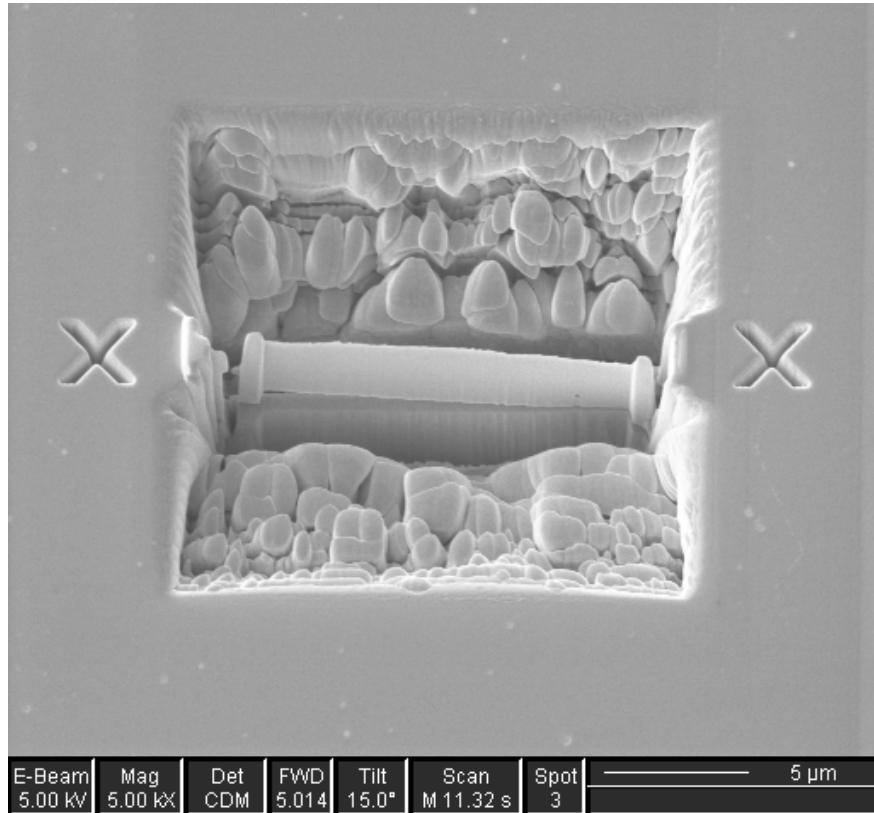
Heat treatment	T_c (K)		B_{c2} (T)	
	25at%Al	22.7at%Al	25at%Al	22.7at%Al
800°C/10h	16.50	16.80	23.93	23.55
furnace heating to 1000°C in 30min + 800°C/10h	17.05	17.05	26.00	24.44
insert in furnace at 1000°C, hold for 2min + 800°C/10h	17.65	17.50	27.49	26.43
ohmic pulse 1000°C/30s + 800°C/10h	17.85	17.65	27.61	26.78
A15-quenched + 800°C/10h	18.20	--	28.54	--

XRD peak broadening



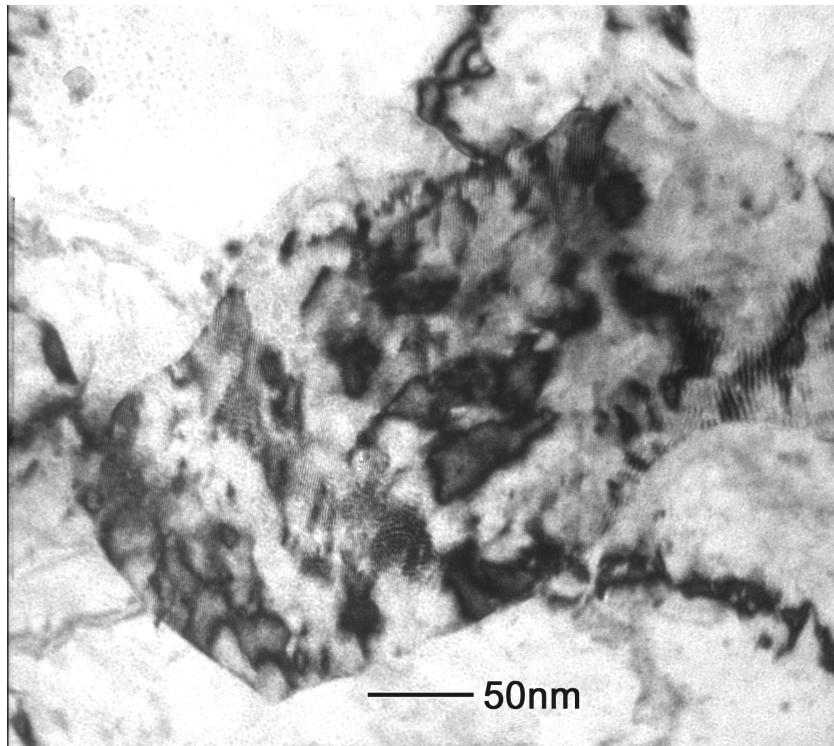
- bcc-quenched + 800°C/10h
- bcc-quenched + furnace heating to 1000°C in 30min + 800°C/10h
- bcc-quenched + insert in furnace at 1000°C, hold for 2min + 800°C/10h
- bcc-quenched + ohmic pulse 1000°C/30s + 800°C/10h

TEM sample preparation



Focused ion beam milling, lift-out technique.

TEM on sample transformed at 800°C/10h



Hundreds of nanometers grain size.

Summary

- Jelly-roll laminate size affects critical current density.
- Optimal maximum temperature at rapid heating found to be in a range just above the temperature of the bcc formation reaction.
- At stoichiometric composition the bcc solid solution forms by a eutectoid reaction between A15 and σ -phase.
- At the other compositions investigated the A15 and the σ -phase, respectively, transform massively to form bcc solid solution.
- This phase evolution cannot be explained based on the published equilibrium phase diagrams.
- Superconducting properties were found to improve as the initial heating rate in the transformation heat treatment increases.
- Increased peak broadening in XRD patterns could be attributed to larger number of planar defects. Mechanism of formation to be investigated.